A STUDY ON THERMAL CONDUCTIVITY OF SIX UNEXPLORED NIGERIAN CLAYS FOR POSSIBLE REFRACTORY AND INSULATING MATERIALS

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DEDICATION

This thesis is dedicated to my beloved family; in loving memory of my late father late Engr. Festus Abara Amkpa and my late mother Mama Rakiya Rabecca Amkpa for their prayers. The thesis is also dedicated to my dearly wife Blessing Jummai Amkpa and my children Victor Akamsoko Amkpa, Victoria Rakiya Amkpa and Vincent Ayetum Amkpa for their prayers, patience, sacrifices and most of all their understanding throughout this academic journey.

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ABSTRACT

Refractories are mineral and chemical based materials, with high heat resistance properties. Refractory manufacturing typically uses clay as the main raw material. The study aims to investigate six unexplored Nigerian clay based on the suitability of their chemical, physical, mechanical and thermal properties as refractories. Six clay deposit locations in Nigeria were selected namely Kpata, Riyom, Gombe, Aloji, Barkin-lade and Quan’pan. The samples were obtained through a two meters depth excavation method. Experimental specimens were produced from these samples through dry-pressing methods followed by firing at 900-1200°C. All specimens were tested to identify the chemical, physical, mechanical and thermal properties of ASTM standards. X-ray fluorescence (XRF) and X-ray diffraction (XRD) analysis show that all specimens contain alumino-silicates as the dominant composition, with 10% loss on ignition. Through the Archimedes test, the percentage of porosity is found in the range of 20-30%, with bulk densities about 1.7-2.3g/cm^3. Cold crushing strength (CCS) and modulus of rupture (MOR) give readings of 15-59 MPa and 6.2-9MPa respectively. Meanwhile, the analysis on thermal properties found specimens having thermal shock resistance on a 20-30 scale cycle, pyrometric cone equivalence (PCE) at a temperature range of 1500-1700°C and thermal conductivity at a range of 0.01-0.9W/m.K. The overall experiment results are within the range of ASTM standards and this illustrates that all of the clay is potentially a refractory material.

ABSTRAK

Refraktori adalah bahan-bahan berasaskan mineral dan kimia, dengan sifat kerintangan haba yang amat tinggi. Pembuatan refraktori lazimnya menggunakan tanah liat sebagai bahan mentah utama. Kajian ini bertujuan menyelidik enam tanah liat Nigeria yang belum diterokai berdasarkan kesesuaian sifat kimia, fizikal, mekanik dan terma masing-masing sebagai refraktori. Enam lokasi deposit tanah liat di Nigeria telah dipilih iaitu Kpata, Riyom, Gombe, Aloji, Barkin-lade and Quan'pan. Sampel-sampel tersebut diperolehi menerusi kaedah penggalian sedalam dua meter. Spesimen ujikaji telah dihasilkan daripada sampel-sampel tersebut menerusi kaedah penekanan kering dan disusuli dengan pembakaran pada suhu 900-1200°C. Kesemua sampel ini diuji bagi mengenalpasti ciri-ciri kimia, fizikal, mekanik dan terma berasaskan piawaian ASTM. Analisis pendafluor sinar-X (XRF) dan pembelauan sinar-X (XRD) menunjukkan bahawa kesemua spesimen mengandungi alumino-silikat sebagai komposisi dominan, dengan 10% kehilangan pada nyalaan. Menerusi ujian Archimedes, peratus keliangan pula didapati dalam julat 20-30%, dengan ketumpatan pukal sekitar 1.7-2.3g/cm^3. Ujian kekuatan hancur sejuk (CCS) dan modulus pecah (MOR) masing-masing memberikan bacaan 15-59 MPa dan 6.2-9MPa. Sementara itu, analisis ke atas sifat terma mendapati spesimen mempunyai rintangan kejutan terma pada skala 20-30 kitaran, kesetaraan kon pirometrik (PCE) pada julat suhu 1500-1700°C dan kekonduksian terma pada julat 0.01-0.9W/m.K. Keseluruhan keputusan ujikaji adalah di dalam julat piawaian ASTM dan ini menggambarkan bahawa semua tanah liat tersebut berpotensi sebagai bahan refraktori.

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LIST OF SYMBOLS AND ABBREVIATIONS

A (m^2)	Cross section area of heat flow in a material
AP	Apparent porosity
ASTM	American Society for Testing and Materials
AU	Gold
Avg	Average
B	Breadth
BD	Bulk density
CCS	Cold crushing strength
CTE	Coefficient of Thermal Expansion
D (m)	Diameter
DSC	Differential Scanning Calorimetry
DTA	Differential Thermal Analysis
E	Total energy
EDX	Energy Dispersive Spectroscopy
F	Force (N)
J	Joules
j/g°C	Specific heat
kg	Kilogram
L	Length
m	Mass of the of the clay brick
MOR	Modulus of rupture
MPa	Mega Pascal
PCE	Plyometric Cone Equivalent
PFS	Percentage firing shrinkage
Q/A	Heat flux
q	Heat flow

S	Soaked weight
SEM	Scanning Electron Microscopy
SG	Specific gravity
STA	Simultaneous Thermal Apparatus
T	Temperature (°C)
TES	Thermal energy storage
TGA	Thermogravimetric Analysis
TPS	Transient plane source
W	Width (mm)
W/mk	Thermal conductivity
PWA	Percentage Water absorption
XRD	X-ray Diffraction
XRF	X-ray Fluorescence
$\Delta T/\Delta L$	Thermal gradient
μm	Micrometer
ΔL	Change in Length
ΔT	Change in Temperature
ρ	Density, g/cm^3

CHAPTER 1

INTRODUCTION

1.1 Background

Heat recovery systems in the early human endeavors used water as a storage medium (Abhat *et al*., 1978). Therefore, solar energy source and household demands for it in general, did not match each other at any given time. This trend, have necessitated the use of thermal energy storage (TES) systems to resolve the mismatch so as to provide energy requirements (Abhat, 1983). Obviously, the potentials are huge for the application of thermal energy storage systems for our homes and industries. TES systems can facilitate an important role, as they provide great potential for facilitating energy savings and reduce environmental impact (Ibrahim and Rosen, 2002). These systems of thermal energy storage were not used as it should and was due to many reasons. Most of these systems are not yet economically competitive with fossil fuels and their long term reliability is not yet ascertained.

It is very necessary to search for an alternative means by the use of thermal insulators around such storage systems in order to maintain high temperatures inside by preventing heat losses to the surroundings (Turner and Malloy, 1988). There are different insulating materials which come in various forms like loose fill rigid boards, pipe and insulating form. Proper and adequate selection of the insulating materials to be used is based on the thermal properties which include the thermal conductivity, specific heat capacity and thermal diffusivity.

The thermal insulation is provided by embedding insulation materials at least on the roof top areas of the furnace and vertical walls of the storage system (Novo *et al.*, 2010). When thermal insulation of the heat storage system is poorly done, it leads to high heat losses (Bauer *et al.*, 2010). Currently fibre glass and rock wool are used as thermal insulation materials for the storage systems. Basically it is due to their low thermal conductivity values leading to good thermal insulation. These thermal insulators however contained siliceous and hazardous dust whose inhalation will increases the danger for the development of lung disease Gilham *et al*. (2016) and are expensive and also dangerous to human health as a result of exposure during handling especially those in fibrous form (Bardelli *et al.*, 2017).

Previous studies have shown that people who manufacture fibre glass have sixty percent more fibre glass material in their lungs than those who had not been exposed (Merler *et al.,* 2017). There is the need for finding alternative thermal insulating materials which are processed from clay which are cheap, reliable and do not pose a risk to human health, for example, kaolin fabricated into aluminium silicates (Al-Malah and Abu-Jdayil, 2007).

Thermal insulators are materials or combination of materials which are used in order to retard the flow of heat energy. The effective installation of thermal insulation can significantly reduce the thermal energy lost from thermal heat storage system surfaces. The energy lost for an insulating material depends largely on the thermal properties and thicknesses of the insulation. The choice of the type and form of the proper insulation materials depends on where the insulation is to be applied as well as the desired material's physical and thermal properties (Al-Homoud, 2005).

The basic requirement for thermal insulation is to provide a significant resistance path to the flow of heat through the insulation material. In accomplishing this, the insulating material must reduce the rate of heat transfer by conduction. In the experimental determination of the thermal conductivity of solids, a number of different methods of measurement are required for the different ranges of temperature and for various classes of materials having different ranges of thermal conductivity values (Aksoz *et al.*, 2012).

The knowledge and concept of heat transfer in insulating material or porous media has increasingly found relevance in science and engineering (Akinyemi *et al.*, 2011). Similarly, Sauer *et al.* (2003), have expressed that thermal properties of porous insulating material as of great importance to environmental sciences, agriculture and engineering, especially in relation to temperature and energy transfer which is better understood in the study of thermal conductivity of materials, its measurement and predicting how much heat can be stored is a key to its utilization by the industries.

The increasing demand for high refractory materials to work on other at high temperatures coupled with the over reliance on the imported refractory has presented a rethink for the technological and industrial development of Nigeria. It is either Nigeria discontinue importation of these refractory bricks and develops its own technology or become import dependence with retrogressive economic implication. The raw materials for the manufacture of fireclay refractory largely contained alumina, silica and impurities. These raw materials deposits are found in very large commercial quantities all over the six geopolitical zones of Nigeria. The clay specimen used in this research were collected from two geopolitical zones that comprises of three states of Gombe, Plateau and Kogi as presented in Figure 3.2 Nigerian Map indicating the clay samples deposit sites.

At the moment, the country depends largely on foreign insulating and refractory bricks even though, reviewed literatures have shown that these raw materials are dominate in its location and ground. Literatures have indicated some scanty works on Nigerian clays in an attempt to understand their properties, but it was not sufficient enough. The investigation of differient clays without their knowledge of thermal characteristics leave so much to be desired. The clay raw specimen when properly studied or investigated, the results can be utilized in manufacturing refractories, other engineering products and by so doing encourage local industrial development in Nigeria (Obadinma, 2003; Yakubu and Abdulrahim 2014).

Table 1.1: Clay deposits and District

Description	Deposit	District	Local Geology	Reference
A	Kpata	Bassa local government area, Kogi East (Kogi State)	Tropical in nature with dry and we seasons. Primary deposits of kaolin are generally formed by the alteration of alumino- silicate rich parent rock such as granite by weathering. Rocks which comprises of the various grouping of coarse grained granites	Odigi, (2000); Imasuen *et al.* (2009), Imasuen *et al.* (2013)
B	Riyom	Riyom local government area, Jos south (Plateau State)	The Jos Plateau state has the highest mineral deposits in Nigeria which are in large quantity and among which are tin, coal, dolomite, kaolin, feldspar, calcium, iron ore, bauxite, tantalite and barite. The Riyom town is situated in the tropical zone.	Hassan *et al.* (2015)
C	Gombe	Gombe local government area, Gombe (Gombe State)	Gombe is blessed with minerals in commercial quantity which includes uranium, gemstone, clay, feldspar, gypsum, kaolin, mica and limestone. It belongs to the tropical zone with wet and dry seasons.	Mbaya *et al.*(2012)
D	Aloji	Ofu local government area Kogi East (Kogi State)	The soil within the clay deposit site is generally loamy having composed of silt, sand and clay. Kaolin deposits are generally located in this site as either primary (residual) or secondary (sedimentary). The mode of formation of the kaolin may have considerable influence on the mineralogy. Tropical	Odigi, (2000); Imasuen *et al.* (2009), Imasuen *et al.* (2013)
E	Barkin-ladi	Barkin-ladi local government area Plateau South (Plateau State)	There are also sizeable pockets of loamy soil of volcanic origin in the high Plateau. Currently in Barikin-ladi, minerals that are mined and processed in commercial quantity are the kaolin, tin, coal, dolomite, tantalite, clay, quartz and calcium. It temperate climate.	Hassan *et al.* (2015)
F	Quan'Pan	Plateau South (Plateau State)	It has a temperate weather with temperature between 18-22 °C. Dolomite, coal, quartz, kaolin, clay and tin is still mined in large commercial quantity and processed on the plateau.	Hassan *et al.* (2015)

According to Manukaji (2013) and Abuh *et al*. (2014) that there is no state in Nigeria without a clay deposit and not many of them have been thoroughly evaluated thermally and utilized for industrial purposes. The thermal conductivity measurement and other thermal properties analysis of refractory materials from Nigeria raw clay will promote but also enhance further studies of the clay abundance and help not to relay on importation of refractories Atanda *et al*. (2013) and this will certainly bring the much needed local industrial development.

Thermal conductivity is simply the measure of a material to conduct heat through its mass. Different insulating materials and other type of material have specific thermal conductivity values that can be used to measure their insulating effectiveness. Li *et al.* (2012) defined thermal conductivity as the amount of heat or energy (K) that can be conducted in unit time through unit area of unit thickness of material, when there is a unit temperature difference.

As indicated by Tiwari *et al.* (2013), there has been an increased awareness of the importance of accurately calculating the thermal conductivity of refractory and heat-insulating materials for furnace design, these values fluctuate considerably depending on the method and the measurement conditions. In their submission, Katsube *et al.* (2006), said that recent urgent demands for reduced energy consumption and efficient energy usage require high performance thermal insulation materials. Such demands have been made in the field of refractory materials. Conventional insulating brick and refractory bricks have good heat-resistance performance and can be produced at low cost (Shimizu *et al.*, 2013) relatively with poor thermal insulating performance.

Schulle and Schlegel (1991) opined that research towards a better understanding of the physical properties of heterogeneous solids has both scientific and technological importance. Similarly, Kingery (1960) discussed that physical properties that determine much of the utility of ceramic (refractories) materials are those properties directly related to temperature changes. Litovsky *et al.* (1996) posited that particular class of these solids is constituted by materials containing a large volume fraction of porosity which are used in situations requiring very good thermal insulation.

Grandjean *et al.* (2005), expressed that prediction of their thermal properties and especially the effective conductivity by analytical or computer calculation is therefore of strong interest. A specific investigation by Aksoz *et al.* (2012) indicated that thermal conductivity using the transient method is calculated from the thermal diffusivity with a further knowledge of the density and specific heat of the materials. This research seeks to investigate the methods of measuring thermal conductivity using the steady state method of the selected clay specimen from Nigeria and their thermal properties in an attempt to encourage its uses in the engineering practices in Nigeria. The properties with which the research was mainly concerned about are the chemical composition; physical (porosity, bulk density, firing shrinkage, water absorption, and specific gravity of the refractory material), mechanical (cold crushing strength and modulus of rupture), thermal (thermal conductivity, heat capacity, coefficient of thermal expansion at room temperature of all the clay deposits specimen. This was further collaborated by Kingery (1960), that a theory and practice to arrive at the heat capacity and thermal conductivity to determine the temperature changes in a refractory/insulating material should be a welcome idea.

1.2 Problem statement

Recent urgent demands for reduced energy consumption and efficient energy usage require high performance thermal insulation materials (Katsube *et al.*, 2006). Such demands have been made in the field of insulating materials and refractories. At the moment it is often not enough getting approximate data from textbooks, but thermal measurements of materials are necessary and the rapid technology development for decades have generated an increasing effort to expand our knowledge of thermal properties (Papadopoulos, 2005). The uses of refractory and insulating materials that can withstand high temperature without deformation and for the prevention of heat losses (escape) respectively, in exchangers, boilers, reactors, ovens, and furnaces in industries where there application are required like in Nigeria and other parts of the world is the main concern. The results are analyzed using scientific methods. The findings are presented in an overview of the thermal properties and their performance characteristics. The main features of measurement and analysis of thermal conductivity of the clay and its applications in thermal energy storage should be studied to match the requirement of the industries because of its low thermal conductivity.

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